INTERNATIONAL STANDARD

ISO 16751

First edition
2020-06

Soil quality — Environmental availability of non-polar organic compounds — Determination of the potentially bioavailable fraction and the non-bioavailable fraction using a strong adsorbent or complexing agent

Qualité du sol — Disponibilité environnementale des composés organiques non polaires — Détermination de la fraction potentiellement biodisponible et de la fraction non biodisponible en utilisant un agent adsorbant fort ou un agent complexant

Reference number
ISO 16751:2020(E)

© ISO 2020



COPYRIGHT PROTECTED DOCUMENT

© ISO 2020

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword ..... **iv**
Introduction ..... **v**
1 Scope **1**
2 Normative references **1**
3 Terms and definitions **1**
4 Principle **2**
5 Reagents **3**
6 Apparatus **3**
7 Procedure **4**
7.1 Sample preparation 4
7.2 Determining water content 5
7.3 Method A: Cyclodextrin 5
7.3.1 Extraction of the sample 5
7.3.2 Phase separation 5
7.3.3 Extraction from receiver phase 5
7.4 Method B: Tenax® 5
7.4.1 Extraction of the sample 5
7.4.2 Phase separation 6
7.4.3 Extraction from receiver phase 6
7.5 Measurement 7
7.5.1 Potential bioavailable fraction 7
7.5.2 Non-bioavailable fraction 7
7.6 Blank test 7
8 Calculation **7**
8.1 Potential bioavailable fraction 7
8.2 Non-bioavailable fraction 8
9 Expression of results **8**
10 Validation **9**
11 Test report **9**
Annex A (informative) **Preparation and regeneration of Tenax®** **10**
Annex B (informative) **Illustrations** **11**
Annex C (informative) **Validation study results** **14**
Bibliography **25**

© ISO 2020 – All rights reserved

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 7, *Soil and site assessment*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

© ISO 2020 – All rights reserved

Introduction

The solubility of most non-polar organic contaminants is limited and they are sorbed to the soil matrix. They may desorb and become available for organisms, which may result in an effect (toxicity, degradation or bioaccumulation). Not all sorbed (adsorbed and absorbed) contaminants will desorb and become available.

Extractions used in chemical analysis to measure the total concentration, release more contaminants from the soil than are available. It is however also possible that contaminants are so strongly bounded by the soil that they will not be released by chemical extraction. This strong sorption may also be caused by incorporation of the contaminant (or a degradation or reaction product of the contaminant) in the organic soil structure. The distribution of contaminants over sorption sites of varying sorption strength is not constant in time and contaminants will shift, with increasing contact time, to the stronger sorption sites.

Figure 1 shows schematically the differentiation between:

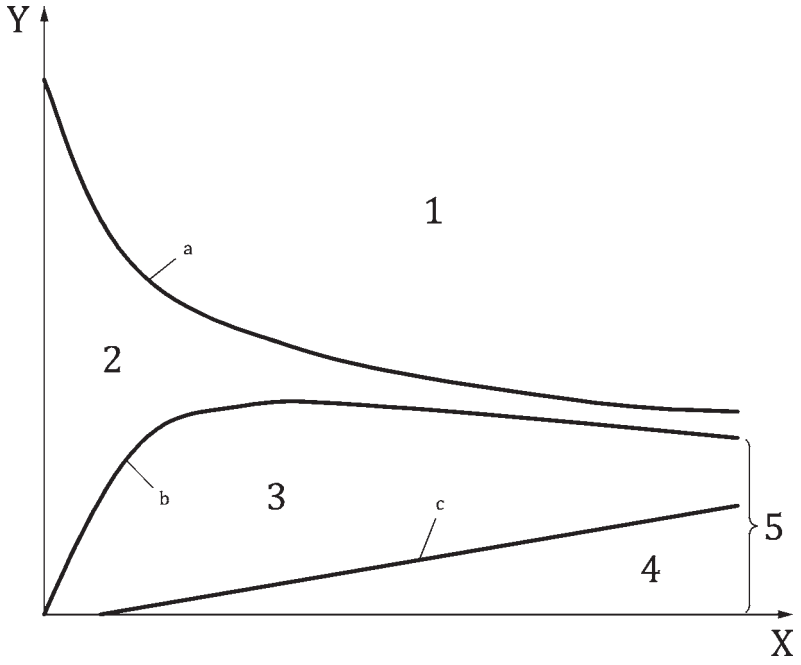
— extractable residues that are also bioavailable (i.e. the potentially bioavailable fraction);

— residues that are extractable by harsher extraction methods but are non-bioavailable;

— residues that are neither extractable nor bioavailable.

If a degradable substance enters a soil, part of it will degrade over time (curve a). The area between curve a and c is extractable by exhaustive chemical procedures. For risk assessments, this part is considered as the “total concentration” for which values are defined in many regulations. However, only a part of this amount is bioavailable. The area between curves a and b is the bioavailable fraction and the area between curves b and c is the non-bioavailable fraction. The method described in this document enables the measurement of the potential bioavailable and the non-bioavailable fraction of a contaminant in soil.

© ISO 2020 – All rights reserved



Key

X time
Y contaminant concentration
1 degradable
2 bioavailable
3 extractable, non-bioavailable
4 non-extractable: persistent residues
5 non-available fraction

NOTE For curves a, b and c see description in text above.

Figure 1 — Temporal changes in extractable/bioavailable fractions, extractable/non-bioavailable fractions and non-extractable/non-bioavailable fractions of a non-polar organic contaminant (modified from [1])

In the scientific research to bioavailability a large number of definitions and concepts are in use, which reflect the discussion in the scientific world. However, a more clear and simple approach is necessary. In regulation, organic contaminants are either bioavailable or non-bioavailable. To support decisions, both should be measurable. Therefore, this document follows the approach of Ortega-Calvo et al. (2015) [2] as illustrated in Figure 2. In this approach all defined fractions are measurable as further explained in Clause 4.

© ISO 2020 – All rights reserved

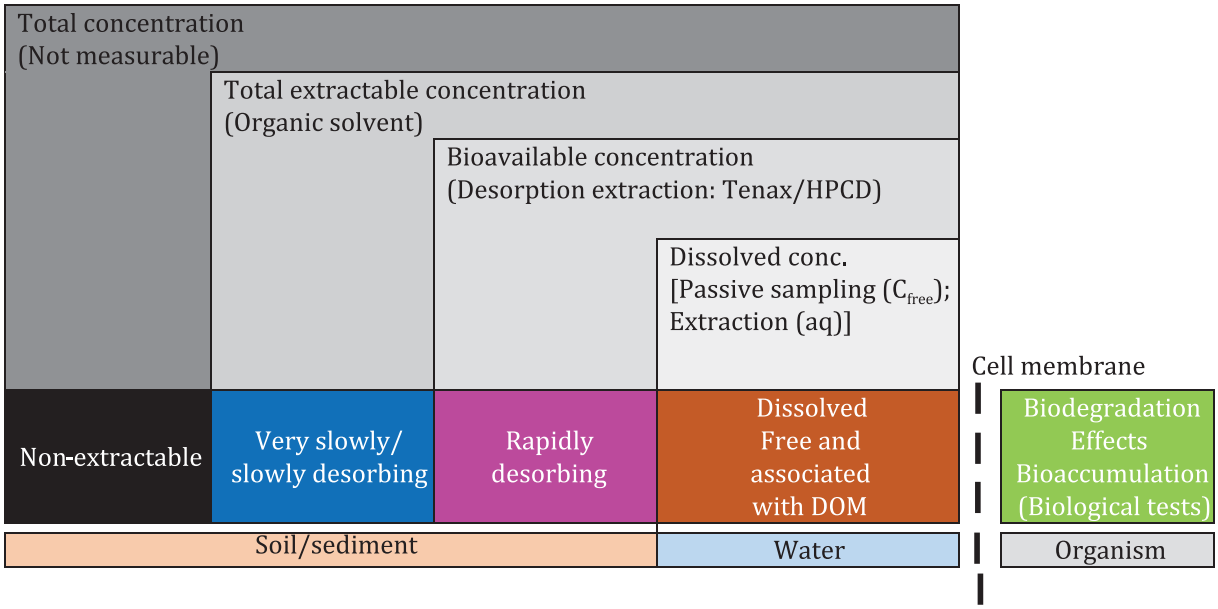


Figure 2 — Measurement of bioavailability of organic chemicals: a simplified scheme for use in regulation[9]

The colour boxes at the left of the biological membrane represent the distribution of pollutant molecules among four classes (non-extractable, very slowly/slowly desorbing, rapidly desorbing and water-dissolved) in soils and sediments. In the scheme in Figure 2, the bioavailable chemical is represented by the rapidly desorbing and dissolved concentrations. The chemical methods able to measure the pollutant present in each specific fraction are given in the grey boxes. The green box to the right of the cell membrane represents the processes that occur within the organism exposed to the pollutant. These biological processes can also serve as the basis for standard methods used for bioavailability measurements.

As presented in Figure 2, the bioavailable fraction can be measured using the method described in this document.

© ISO 2020 – All rights reserved

Soil quality — Environmental availability of non-polar organic compounds — Determination of the potentially bioavailable fraction and the non-bioavailable fraction using a strong adsorbent or complexing agent

1 Scope

This document specifies an extraction method to determine the bioavailable (potential and environmental available) fraction and the non-bioavailable fraction of a contaminant in soil using a "receiver phase" for an organic contaminant with strong sorbing or complexing properties, for example, Tenax®[1)] or cyclodextrin, respectively.

NOTE 1 The bioavailable fraction is defined in ISO 17402 as environmental bioavailability.

The method is applicable for non-polar organic contaminants with an aqueous solubility of <100 mg/l. The method is applicable for soil and soil-like material including (dredged) sediments.

NOTE 2 The method is theoretically applicable to non-polar organic contaminants with an aqueous solubility of 1 000 mg/l. The method has been often applied for compounds with a much lower solubility (K_{ow} > 3) and less for compounds with a higher solubility. The applicability is therefore defined for compounds with an aqueous solubility of <100 mg/l.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 11074, *Soil quality — Vocabulary*

ISO 11465, *Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method*

ISO 14507, *Soil quality — Pretreatment of samples for determination of organic contaminants*

ISO 18512, *Soil quality — Guidance on long and short term storage of soil samples*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11074, ISO 17402 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

1) Tenax® is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

© ISO 2020 – All rights reserved

3.1
potential bioavailable fraction
amount of contaminant present in the matrix that can be released from the solid phase to the aqueous phase in a well-mixed water soil mixture and in presence of a receiving phase in a period of 20 h

Note 1 to entry: In ISO 17924 the term bioaccessibility is used, which is the fraction of a substance in soil or soil-like material that is liberated in (human) gastrointestinal juices and thus available for absorption. This document does not distinguish between bioavailability and bioaccessibility and uses the general term bioavailability. The concept of bioavailability as followed in this document is described in the introduction of this document.

4 Principle

This method gives an estimation of the potential bioavailable and non-bioavailable fraction of organic contaminants, i.e. the amount of the contaminant in the matrix that is potentially exchangeable with the aqueous phase; specifically that, which is adsorbed/complexed by Tenax®/cyclodextrin.

The extractable and non-bioavailable fraction of the contaminant left in the sample following the action of Tenax®/cyclodextrin can be subsequently measured with an exhaustive/harsh extraction technique (designed to measure the total concentration) and in this way the non-bioavailable fraction of the contaminant is assessed.

Thus, in numerical terms, the total contaminant concentration in a sample is the sum of the bioavailable concentration (established using a strong sorbent or complexing agent) and the non-bioavailable concentration (established using a subsequent harsh extraction method performed on the residue that is left after the matrix has been extracted using a strong sorbent or complexing agent):

$$c_{\text{tot,cont}} = c_{\text{bio}} + c_{\text{non-bio}} \quad (1)$$

where

$c_{\text{tot,cont}}$ is the total contaminant concentration;

c_{bio} is the bioavailable concentration;

$c_{\text{non-bio}}$ is the non-bioavailable concentration.

The soil, soil-like material or sediment sample with particle size <2 mm is extracted with water containing a "receiver phase" for the organic contaminants. This phase is either a complexing agent (cyclodextrin) or a strong adsorbent [Tenax®]. The solubility of non-polar compounds is limited and in this method the receiver phase acts as an "infinite sink". The measured amount, which is the amount that desorbs from the soil or soil-like material during 20 h, reflects the fraction of contaminant that can have effects on biotic systems and that can become mobile.

In the following step, the contaminants adsorbed are extracted from the receiver phase and determined by appropriate analytical methods. The amount of contaminants left in the soil residue, the non-bioavailable fraction, can be measured using a subsequent harsh/exhaustive extraction designed to measure the total concentration. Formula (1) can then be used to determine the total contaminant concentration in the sample (if desired).

NOTE 1 ISO 13859 and ISO 13876 are examples to measure the total concentration of respectively PAH and PCB in soil and soil-like materials.

NOTE 2 Formula (1) shows the relationship between the "total contaminant concentration" the "bioavailable concentration" and the "non-bioavailable concentration". If two of the concentrations are known the third concentration can be calculated through the use of Formula (1). For example, by measuring the "total concentration" and the "non-bioavailable concentration", the "bioavailable concentration" can be calculated. This is allowed with homogeneous materials. If it is not known whether a material is homogeneous and the bioavailable concentration is the concentration of interest, the bioavailable concentration needs to be measured.

© ISO 2020 – All rights reserved